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PRODUCTION OF RUTIN FROM BUCKWHEAT LEAF MEAL

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The authors wish to acknowledge the pioneer work of J. F. Couch, who initiated the studies on rutin at this Laboratory, developed the first practical extraction process for green buckwheat, and extracted rutin from dry buckwheat, tobacco, and other rutin-bearing plants. Acknowledgment is also made to C. F. Krewson for consultation and suggestions and to B. A. Brice, M. J. Copley, J. Naghski, and W. L. Porter for development of analytical methods making possible the control of the work. Credit is due Rita Hurley for assistance in drying buckwheat and compiling data.

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SUMMARY

Based on pilot-plant experience, a method is given for the large-scale preparation of buckwheat leaf meal. Such meal can be stored for at least 1 year without deterioration and can be used as a source of the new drug rutin. The necessary equipment and details of operation for two methods of preparing pure rutin from buckwheat leaf meal are described, and operating cycles are suggested. The processes described have been simplified through pilot-plant research conducted since the issuance of AIC-114 in April 1946.

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INTRODUCTION

In April 1946 there was issued AIC-114 "Production of Rutin from Buckwheat Leaf Meal" covering pilot-plant research and development of new processes for the preparation of leaf meal and of pure rutin from it. Since the issuance of this circular, further research on a pilot-plant scale has resulted in improvements in the methods for separating the leaf fraction from the stems of fractionally dried buckwheat and also in the methods for extracting and purifying rutin by the hot-water and the dilute-alcohol processes. The present publication constitutes a revision of AIC-114 and supersedes it.

As a result of J. F. Couch's suggestion in 1941 that rutin might reduce capillary fragility, clinical tests were made by J. Q. Griffith, Jr.[4] These tests established the value of rutin for this purpose.

Couch originally obtained rutin from high-grade tobacco,[5] but the high cost of this raw material impelled him to search for other sources. Buckwheat was found to be an economical source. He obtained pure rutin by a process involving the extraction of fresh buckwheat with alcohol.[6]

PREPARATION AND STORAGE OF BUCKWHEAT LEAF MEAL

Buckwheat Harvesting

Studies by J. F. Couch[6] showed that the percentage rutin content (on a moisture-free basis) of the entire Japanese buckwheat plant (*Fagopyrum esculentum*) reaches a maximum at an early stage of growth, when only a few blossoms are yet formed. As the growth of the plant proceeds, the percentage of rutin decreases, owing in part to the relatively faster growth of the stem tissues, which contain a much smaller percentage of rutin. We may assume that the percentage of rutin in the leaves and flowers, and thus in the dried leaf meal, remains approximately constant until the blossoms begin to change into seeds or the leaves begin to atrophy.

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[4] J. Q. Griffith, Jr., J. F. Couch, and M. A. Lindauer. Effect of Rutin on Increased Capillary Fragility in Man. Proc. Soc. Exptl. Biol. and Med., 55, 228-9, 1944.

[5] J. F. Couch, and C. F. Krewson. Rutin. United States Department of Agriculture AIC-52. (Eastern Regional Research Laboratory) July 1944.

[6] J. F. Couch, C. F. Krewson, and J. Naghski. Extraction and Refining of Rutin from Green Buckwheat. United States Department of Agriculture AIC-160. (Eastern Regional Research Laboratory) July 1947.

Couch[6] states that the maximum yield of rutin from Japanese buckwheat per acre is obtained when the buckwheat is harvested at full bloom, just before the seeds have set, roughly 28 to 35 days after sprouting. He states further that Tartary buckwheat (*Fagopyrum tataricum*) has advantages over the Japanese variety as a source of rutin. The former contains more rutin, retains its rutin content for a longer time, and has more resistance to frost. This variety reaches the harvest stage between 35 and 50 days after sprouting.

Preparation of Buckwheat Leaf Meal

GENERAL - Couch's findings on the superiority of the Tartary variety of buckwheat were made too recently to permit drying studies on this variety. Hence, the information given here for drying buckwheat and preparing leaf meal from it is based on experience with the Japanese variety only. Furthermore, in the absence of data on the artificial drying of harvested buckwheat which has been allowed to wilt in the field, our calculations and recommendations are of necessity based on drying the buckwheat immediately after harvesting.

Experience has shown that buckwheat leaf meal prepared as described here can be stored in the dark for more than a year without loss of rutin. Our tests included meals having moisture contents between 5.8 and 9.3 percent. It can be safely assumed that a manufacturer can produce rutin steadily throughout the year from meal prepared during the summer.

Drying the leaves and flowers must be rapid to avoid the serious loss of rutin that occurs during slow drying. Couch, Krewson and Naghski[7] found that most of the rutin in tobacco is lost in the air-curing process; the behavior of the buckwheat plant is similar. Laboratory tests of Couch, Naghski, and Krewson[8] showed the large loss of rutin that occurs when the plant is completely dried in a laboratory oven at temperatures ranging from 160° to 230° F. However, since the stems of the plant contain little rutin, they can be discarded without drying. By stopping the drying operation as soon as the leaves become dry and by choosing drying conditions specifically designed to minimize loss, the over-all loss of rutin can be reduced to about 25 percent.

If preferred, the entire buckwheat plant may be dried to prepare a "whole" meal, which may also be used as a source of rutin. The rutin content of such a product will be approximately 75 percent of that of the fresh plant on a dry basis, about 25 percent being unavoidably destroyed in the drying operation. However, the percentage rutin content of whole meal is obviously less than that of leaf meal, because the whole meal includes the stems, which contain only an insignificant amount of rutin. Though the tonnage of whole meal produced would be

[7] J. F. Couch, C. F. Krewson, and J. Naghski. Preparation of Rutin from Buckwheat. Presented at the meeting of the American Chemical Society in Philadelphia on June 13, 1945.

[8] J. F. Couch, J. Naghski, and C. F. Krewson. Buckwheat as a Source of Rutin. Science 103, 197 (1946).

much greater than that of leaf meal from the same amount of buckwheat, the total weight of rutin in it would be no greater. Therefore our work has been largely directed toward determining the optimum conditions for the preparation of leaf meal.

Experience during the past year has indicated that some types of alfalfa driers may be successfully employed in the production of whole buckwheat meal. Belt driers will be adaptable to the production of leaf meal if they can be operated under the conditions described later in this publication.

THE PROCESS - It is assumed that drying the buckwheat plant and preparing leaf meal from it will be done at or near the place of growth and that the meal will be shipped to the rutin manufacturer.

The recommended procedure is to dry as quickly as possible and carry the drying only far enough to embrittle the leaves and flowers; the stems, being much thicker, remain moist and tough. The plants in this condition are subjected to mechanical action, which breaks the brittle leaves and flowers away from the stems and crushes them into fragments. This is accomplished by directing the fractionally dried buckwheat into a fan, which, acting somewhat as a hammer mill, strips the friable leaves from the limp stems. The fan also blows the material to a cyclone collector. From there it falls to a vibrating screen, which separates the stems from the leaf fragments. A bag filter is provided on the cyclone exhaust in order to recover rutin-rich fines. The material in various stages of preparation is shown in Figure 1.

A flow sheet for the preparation of leaf meal is given in Figure 2. Figure 3 shows the drier used in the pilot plant.

EQUIPMENT AND OPERATING PROCEDURE - For the purposes of this publication, we have made the following assumptions as to the capacity of the drying plant, characteristics of the Japanese buckwheat, and operating data. The figure for moisture content of fresh buckwheat was chosen, to be safe, close to the maximum observed. The other analytical figures are observed averages.

Daily output of leaf meal by drier, tons (3-1/4 times the capacity required by either extraction process)	5
Daily consumption of fresh plant by drier, tons	50.5
Harvesting season (approximate)	June 15 to September 30
Days (24 hours) of operation of drier	75
Moisture content of fresh buckwheat, percent	86
Rutin content of fresh buckwheat, moisture-free basis, percent	2.6
Moisture content of discarded stems, percent	25
Moisture content of leaf meal, percent	8
Rutin content of leaf meal, moisture-free basis, percent	3
Loss of dry matter as stems, percent	35

Obviously, most of the moisture in the leaves can be removed by sundrying the whole plant with a great reduction in the size of the drier and in the cost of drying. It has not been established, however, whether this can be done without prohibitive loss of rutin. Moreover, it has not been possible to make pilot-plant fractional drying studies on such field-wilted material. Hence, nothing is known as to the over-all loss of rutin that might be entailed by preceding factory drying with field wilting. The figures given here are for drying the freshly harvested plant, which corresponds to our actual experience. If it is later shown that drying field-wilted material can be accomplished without greatly increased loss of rutin, field wilting should certainly be done, as the reduction in cost of drying will compensate for a certain amount of rutin loss.

Cutting: To enable the buckwheat plant to be loaded easily and uniformly on the drier belts, it should be chopped coarsely. The chopping must be done with a minimum of crushing or bruising. If the chopped plant is sent to the drier within a few minutes, no appreciable loss of rutin occurs, but there must be no long delay or storage of the chopped material. An ensilage cutter, set to produce pieces about 2 inches long, is suitable.

Drying: The most suitable type of drier for drying the plant in the manner described here is one in which moderately heated air is forced at high velocity through a shallow bed of the material supported on a perforated belt, or belts, moving continuously through the heating chamber.

The drying time can be considerably decreased, and the desirable uniformity of the product insured, by turning the plant over after it has been partly dried. This breaks up compacted mats of wilted material within the bed, and moves the lower material to the more favorable top position, where it receives hotter air. In a multiple-belt drier, this is done by dropping the buckwheat from one belt to another beneath it. A three-belt drier is suitable; this turns the material over twice. The belts should be constructed of flexible wire screen (7 to 10 mesh). The air should be uniformly distributed down through the material at a rate of at least 175 cubic feet per minute per square foot of belt. Suitable heating units with controls should be provided to maintain the inlet air at 230° F. Most of the air is reheated and recirculated. The drier should also be provided with an automatic variable-speed feeding-and-spreading device and rotating doffers to break up the mat of material as it drops from one belt to another.

Normally, the second belt is run at one-half the speed of the first, and the third at one-half that of the second, but the speed of each belt relative to the others should be adjustable. The range of speed of the first belt especially should be liberal, as, for best drying results, buckwheats of different moisture contents and different maturities require different treatments on the first belt. For plants in full bloom and containing 81 percent moisture, the initial load on the first belt should be not more than 2 pounds (fresh weight) per square foot; if too deep a layer is used, the buckwheat when wilted by partial drying settles down to a compact mat that obstructs the passage of air. Under normal operating conditions, the air pressure drop through such a bed is about 0.3 inch of water. Younger plants, or in a rainy season plants in full bloom, may easily contain 86 percent moisture. These,

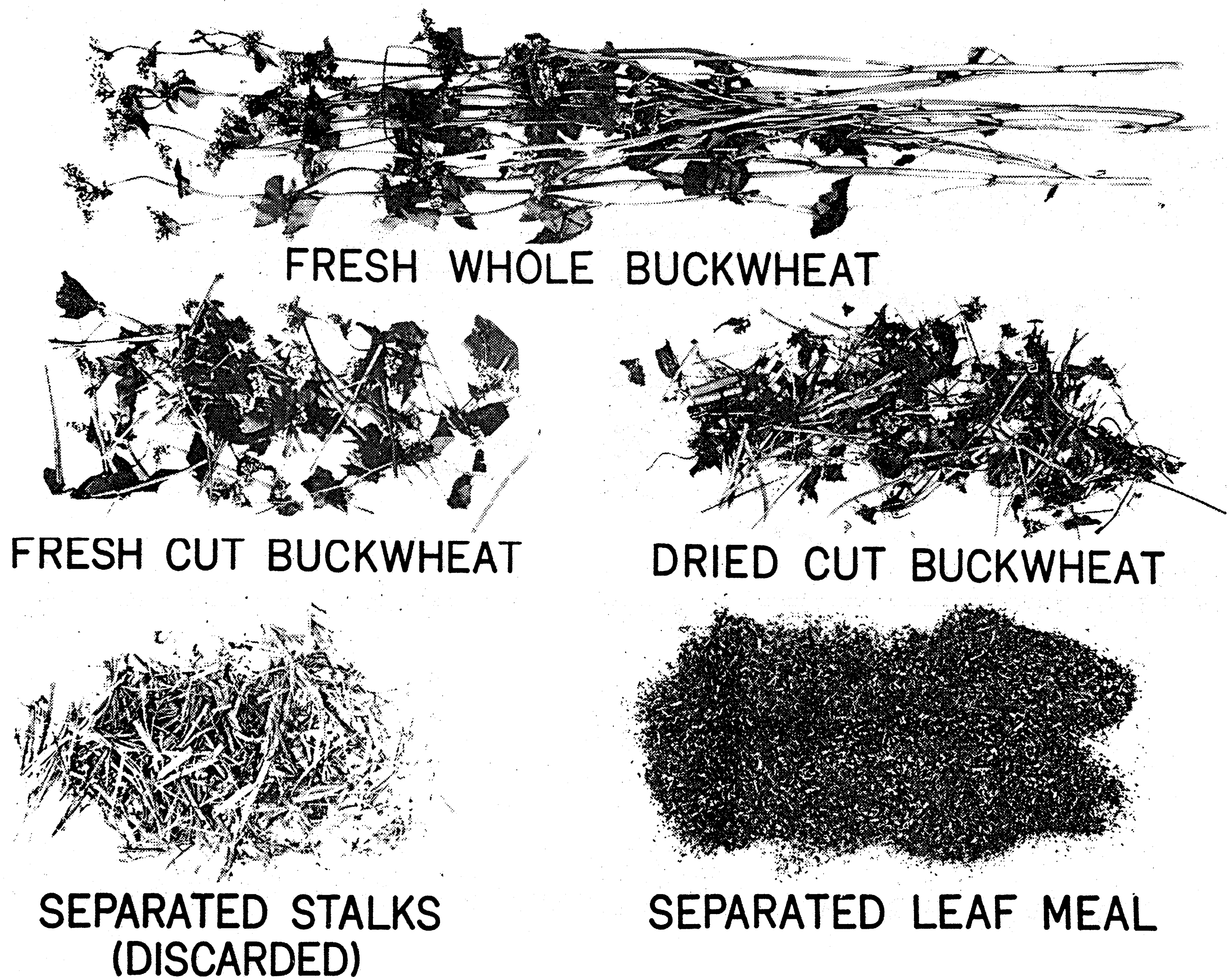
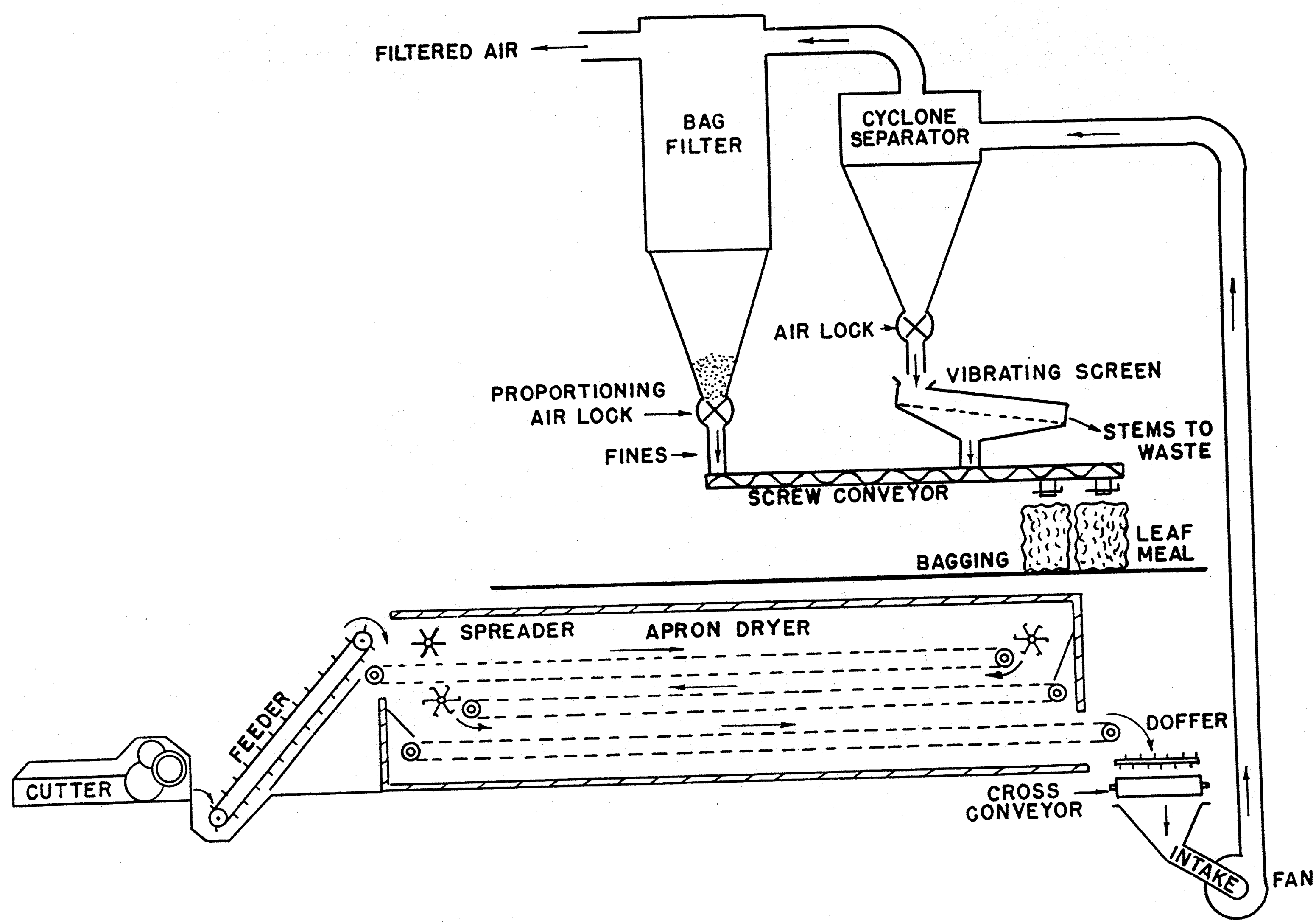


FIG.-1- VARIOUS STAGES IN THE PREPARATION OF BUCKWHEAT LEAF MEAL

FIG. 2 FLOW DIAGRAM FOR PREPARATION OF BUCKWHEAT LEAF MEAL



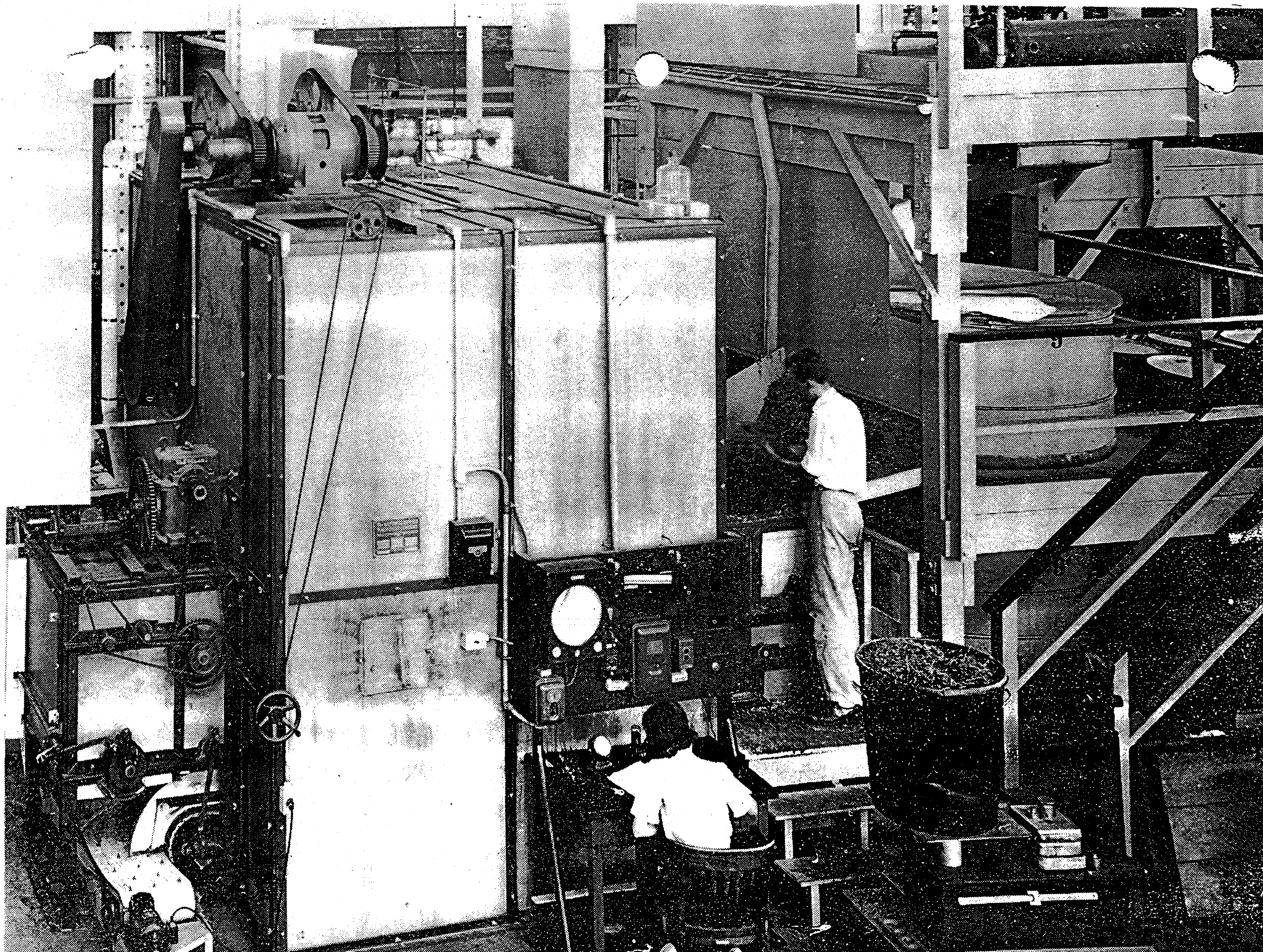


FIG. 3 - PILOT PLANT DRIER

or plants carrying considerable surface moisture, require a lighter load on the first belt, as they are more prone to wilt and form compact mats. This change in load is accomplished by increasing the speed of the first belt relative to the others.

To produce 5 tons of buckwheat leaf meal per day of 24 hours, a drier would have to handle 101,000 pounds of fresh plant of 86 percent moisture content, or 74,400 pounds if the moisture content were 81 percent. We have used the 86-percent figures in calculating the required size of drier, to allow for young plants or a rainy season. On this basis, the belts should have a total drying area of 480 square feet. Three belts 8-1/2 feet wide, each having an active length of 19 feet, would be suitable. This drier could be arranged either with each belt traveling back underneath the preceding one or with all three traveling ahead in the same line. With air at 230° F. dry bulb and 145° F. wet bulb, the total drying time would be about 12.5 minutes but would vary according to the characteristics of the buckwheat. In a drier of these dimensions, this figure corresponds to a speed of about 2.6 feet per minute for the third belt and 5.2 for the second. The first belt should have a range of speed from 6 to 12 feet a minute or thereabouts; if run at 10.4 feet per minute, to handle 101,000 pounds per day the load would be about 0.8 pound per square foot. The drier would probably require about 65 horsepower for fans and drives; if an oil furnace is used for heating, it would consume approximately 50 gallons of oil per hour.

In AIC-114 a drying temperature of 190° F. was recommended. Subsequent experiments have shown that this temperature can be raised to at least 230° F. without increasing the destruction of rutin and with a reduction of about 30 percent in the size of the drier. Our tests on Japanese buckwheat showed an increased loss of rutin at 270° F. The few tests made on Tartary buckwheat indicate that a temperature of 280° F. would probably be satisfactory for this variety.

Separating: To separate the leaf and flower material, now dry and brittle, from the still moist stems, the output of the drier is fed into a flat-blade paddle-wheel fan by means of a cross conveyor and feed hopper. This operation must be carried out immediately; otherwise the moist stems will rehydrate the leaves, and separation will be impossible. A "doffer," or kicking device, is provided on the discharge end of the cross conveyor to break up any mat formations and thereby eliminate the possibility of "bridging" in the feed hopper. With the fan which we used, a tip speed of approximately 10,000 feet per minute was found best. Its diameter was 15-1/4 inches, and it was operated at 2,600 revolutions per minute.

The stripped stems and the leaf fragments are blown by the same fan through a cyclone separator to a single-stage (self-cleaning) vibrating screen having a 5-mesh sieve (openings of 0.159 inch). There the leaf fragments are separated from the stems, and loaded from the sieve outlet into bags. The stems passing over the screen are discarded. A screen area of about 10 square feet should be adequate.

A bag filter is attached to the exhaust end of the cyclone separator to recover the fines, for these are rich in rutin. A motor-driven rotary air lock is provided to proportion the fines back to the leaf meal via a screw conveyor. A total filtering surface of about 700 square feet should be adequate for the bag filter.

SUGGESTED LIST OF EQUIPMENT -

	Cost
Binder-reaper and tractor	$ 2,000
Truck	2,700
Conveyor to cutter	900
Ensilage cutter	650
Drier, with feeder	24,000
Conveyor to fan	225
Fan and cyclone	600
Screen	500
Bag filter with proportioning air lock	1,200
Screw conveyor, with two spouts	350
Stacking conveyor	1,200
Total equipment	$34,325

USE OF CONVENTIONAL ALFALFA DRIERS - Some preliminary field tests were made on drying chopped Japanese buckwheat in a triple-pass, through-circulation, direct heat, rotary drier, but the range of operating conditions investigated was not wide enough to permit final conclusions as to the suitability of such a drier for buckwheat. The highest inlet gas temperature was 1,900° F., and the outlet temperature was 360° F. The lowest inlet temperature was 1,260° F., with a corresponding outlet temperature of 275° F. Intermediate inlet and outlet temperatures were also investigated. The best temperatures of those investigated were the lowest, both inlet and outlet. However, even under these conditions, approximately 50 percent of the rutin in the original buckwheat was lost. It is possible that by further reduction in inlet and/or outlet gas temperatures the buckwheat could be dried without too great a loss of rutin.

As we have already stated, there are indications that in low-temperature driers (200°-300° F.) Tartary buckwheat can safely withstand somewhat higher temperatures than can the Japanese variety. Moreover, using the high temperature drier referred to in the previous paragraph, Porter[9] found that Tartary buckwheat dried at approximately 1,800° F. inlet temperature and at three different outlet temperatures--350°, 375°, and 400° F.--produced leaf meal of 4.0 to 4.4 percent rutin content.

Undoubtedly belt driers other than the triple belt described here could be used satisfactorily for drying buckwheat. The important considerations are (1) that the early stage of drying be accomplished quickly, because destruction of rutin is most rapid while the leaves are in a

9 Unpublished report of work at Eastern Regional Research Laboratory.

moist state, and (2) that the nearly dried plant should not be subjected to high temperature. Thus it may well be that a belt drier with two independently heated stages, a type sometimes used in drying vegetables, tobacco, and alfalfa, could handle buckwheat at lower cost and/or with less loss of rutin than the type described here. Such a two-stage drier could be made to fulfill the two requirements pointed out above. In the first stage, when the buckwheat is moist, a high temperature could safely be employed to shorten the time. In the second stage, a safely low temperature would be used, say 230° F. for Japanese buckwheat or perhaps 280° for the Tartary variety.

HOT-WATER EXTRACTION PROCESS

General Description

In the hot water extraction process, buckwheat meal, either whole meal or leaf meal, is given three short extractions with boiling water. This removes more than 97 percent of the rutin content of the meal. The extracts are filtered and then concentrated by vacuum evaporation. Proteins and colloidal materials are coagulated by adding isopropyl alcohol to the concentrates. The curd so formed is removed by filtration; the filtrate is evaporated, with addition of water, until practically all the alcohol is driven off. This water solution is filtered hot and then permitted to crystallize, and the crude rutin is filtered off and dried. The dried rutin is then dissolved in a small amount of anhydrous alcohol, and the insolubles are filtered off. The alcohol is replaced by water in the course of evaporation; silica gel is added to adsorb red pigment. The hot solution is filtered and allowed to crystallize. The pure rutin is then filtered off and dried.

Figure 4 is a flow diagram for the production of rutin by this process.

Details of Operation

The operations described here are those required in a plant producing 100 pounds per week of pure dry rutin from buckwheat meal containing 3 percent rutin on the moisture-free basis and having a moisture content of 8 percent. For conservatism, a 78-percent yield of pure rutin, based on the rutin content of the meal, is assumed, although in actual pilot-plant practice yields of 80 percent and slightly higher have been obtained. This will produce 5,000 pounds of pure rutin in an operating year of 300 days. The equipment described is that required in a new factory designed for the express purpose of producing rutin. It is recognized, however, that in most cases rutin will be produced in equipment already available in pharmaceutical plants, since much of such equipment will undoubtedly be adaptable to the rutin process. In many cases wooden tanks and wooden filter presses are specified. This represents the cheapest type of equipment that could be employed. When stainless steel equipment is available, it would be preferable. Where stainless steel equipment is specified, it must be used, as less corrosion-resistant materials may contribute impurities to the finished rutin. Even traces of dissolved iron or copper are objectionable.

EXTRACTION - Three extractions are recommended--a 2-hour, a 1-1/2-hour, and a 1-hour. The extraction is carried out in a wooden tank 9 feet in diameter and approximately 7 feet deep, equipped with a false bottom covered with 20-mesh stainless steel screen. Beneath the screen there should be a stainless steel steam sparger, through which the necessary auxiliary heat can be introduced. Into this are charged 285 pounds of buckwheat meal (moisture-free basis) and 20 times that weight (713 gallons) of boiling extractant. Slow-speed mechanical agitation should be supplied to insure intimate mixing, and a small amount of open steam should be introduced to offset radiation losses and insure that actual boiling is maintained throughout the extraction.

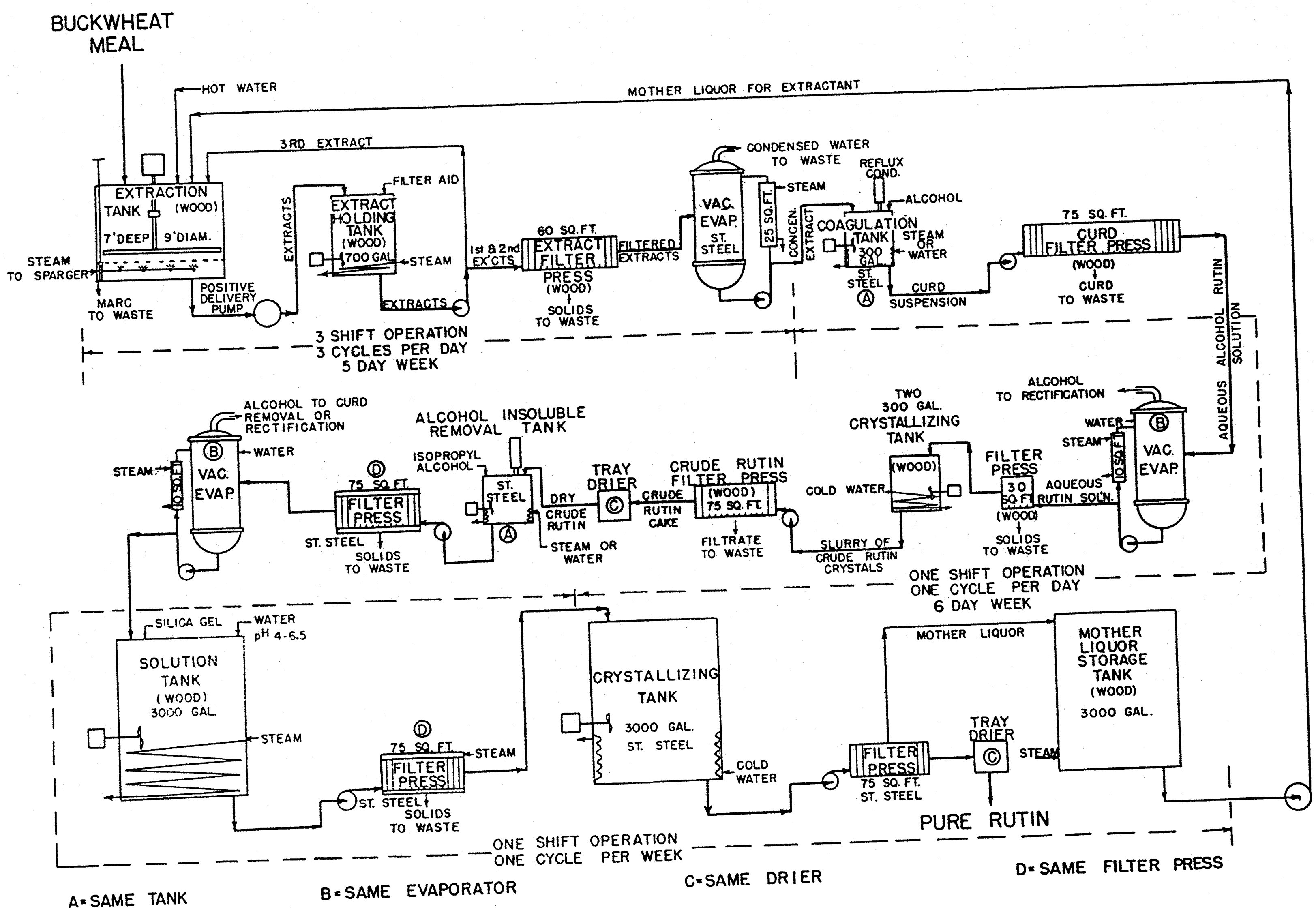
After operations are in progress, the first extraction should be made with the third extract from the preceding batch. This reduces the amount of extract to be evaporated by one-third. The liquid used for the second extraction should be the mother liquor from the final crystallization. This contains a significant amount of rutin, which can thus be in part recovered. Fresh water is used for the third extraction. The pH of the boiling mixture should never be permitted to rise above 7.0. Buckwheat meal is sufficiently acid to insure this in most cases, but when the water is unusually alkaline, a small amount of sulfuric acid may have to be added.

Each extraction should be continued to the point where equilibrium conditions (balanced distribution of rutin between meal and liquor) are obtained. Our pilot-plant work showed that the extraction cycle recommended here achieves equilibrium. Laboratory experiments with much smaller quantities of meal, which could be easily maintained in a state of active ebullition, showed that equilibrium could be reached in less time than that specified here. It is therefore important in designing the extraction tank to insure that good agitation and active boiling will be obtained. Too long an extraction time is not only wasteful of steam but may result in some destruction of the rutin.

The diameter of the extraction tank is important, as there is a limit to the weight of meal that can be loaded per square foot of drainage area and still have rapid and thorough drainage. This load depends on the physical state of the meal and the conditions under which the buckwheat plant is dried. Meals prepared by the method described in this circular may be loaded as high as 9 pounds of meal per square foot. However, many commercial meals are more finely divided and are slow to drain. We have therefore used a figure of 4.5 pounds of meal per square foot of false bottom in the recommended 9-foot diameter tank for handling 285 pounds of meal (moisture-free basis) per batch. This should take care of even the slowest draining meals. Rapid and thorough drainage is important. Slow drainage means slow operation and prolonged maintenance of the extract at elevated temperature. The fraction of rutin removed from the meal in each extraction equals the percentage of the liquor drawn off when equilibrium conditions have been obtained. This should be about 70 percent per extract. For example, in a batch of meal which contains 8.55 pounds of rutin, there would be 5.98 pounds of rutin in the first extract, 1.8 pounds in the second

FIG. 4

FLOW DIAGRAM FOR PRODUCTION OF RUTIN FROM BUCKWHEAT MEAL BY HOT WATER EXTRACTION PROCESS.



extract, and 0.54 in the third extract. These figures are based on the assumption that fresh water is used as the extractant in each case. Naturally, they will be altered somewhat when process liquors are used for the extractants, as recommended above.

With the recommended extraction cycle, a batch of meal can be extracted and the exhausted marc removed from the tank in slightly less than 8 hours. Therefore a total of 930 pounds of meal on an air-dry basis (three 310-pound batches) can be extracted in a 24-hour operation. The exhausted marc is easily removed from the extraction tank if a plug valve outlet is located in the tank so that its opening is flush with the surface of the false bottom. The marc is simply flushed into this outlet with a hose.

CONCENTRATION OF EXTRACTS - Before concentration and the subsequent coagulation step, which employs isopropyl alcohol, the extracts should be filtered to remove finely divided meal, thereby avoiding the extraction of fats from such entrained meal by the alcohol. A wooden plate and frame filter press having about 60 square feet of filtering surface and using 2-inch frames can be employed for this purpose. Fast paper properly backed with cloth for support should be used for dressing the press. A light precoat of filter aid speeds filtration. The press should be preheated with hot water. Ordinarily the first two extracts can be filtered without redressing the press. The third extract is of course not filtered, as it will be used as the first extractant in a succeeding batch.

For flexibility of operation, it is desirable to have a wooden storage tank of approximately 700 gallons capacity between the leaching tank and the filter press. This serves a dual purpose. It permits incorporating about 10 pounds of filter aid into the first extract and 5 pounds into the second to facilitate filtration. This tank also serves as a storage tank for the third extract until it can be used as the first extractant in the succeeding batch. It should be equipped with an agitator and with stainless steel heating coils to keep the solution hot and prevent any crystallization of rutin before filtration.

The extracts are concentrated in a stainless steel vacuum evaporator. If a forced-circulation external calandria-type evaporator is used, 25 square feet of heating surface will be sufficient; if a vacuum pan is used, an increase in the heating surface will be required to achieve the same evaporation rate. Since the first two extracts should be evaporated to about 30 gallons, the evaporator must be capable of reducing approximately 1,050 gallons of extract to 30 gallons every 8 hours, that is, evaporating about 1,020 gallons of water. Evaporation can desirably be carried to the maximum viscosity at which the slurry is still fluid enough to flow from the evaporator. The further evaporation is carried, the less alcohol is required in the subsequent curd-forming step. In most cases, concentration to a reading of 1,000 centipoises, when measured at 107° F. with a Brookfield viscosimeter using a No. 3 bob at 30 revolutions per minute, produces a flowable concentrate.

In order to avoid the possible destruction of rutin through prolonged heating, it is important to carry out the evaporation under a vacuum of at least 26 inches. The extracts from one extraction cycle can be concentrated batchwise. The concentrates from the three extraction cycles carried out over a 24-hour period can be conveniently accumulated in the coagulation tank.

COAGULATION AND REMOVAL OF NONRUTIN SOLIDS - The tank used for coagulation should be made of stainless steel, for it will also be used in a later step, where wood would be unsatisfactory. It should be equipped with a jacket or stainless steel coil into which either steam or water can be introduced, and it should be provided with an agitator and a reflux condenser.

The concentrates accumulated over a 24-hour period are put into this tank, and twice their volume of 91 percent isopropyl alcohol (by volume) is added. Ethyl alcohol should not be used for this purpose. The mixture is brought to the boiling point to insure complete solution of the rutin. Ten minutes' boiling should be sufficient. Long boiling is inadvisable. Cold water is then introduced into the coils of the tank, and the mixture is cooled to about 80° F. It is then ready for filtration.

The curd formed in the coagulation step is removed by filtering in a wooden filter press having about 75 square feet of filtering surface. Two-inch frames should be used to accommodate the curd. A fine twill cloth may be used for dressing the press. Experience has shown that the most rapid filtering is obtained if the pressure is maintained at the lowest practical level and preferably not permitted to rise above about 7 pounds per square inch. If this precaution is observed, the filtration will be rapid, but if the pressure is permitted to rise too high, even for a short time, the filtration rate will be permanently decreased.

In order to obtain the maximum yield of rutin, it is obviously desirable to wash the curd. This can preferably be done by reslurrying it in boiling isopropyl alcohol of about 67 percent by volume, cooling, and then refiltering and adding the wash alcohol to the main batch.

The filtrate from the filter press passes to a stainless steel evaporator, where the alcohol is driven off and replaced with water, evaporation being carried to a final vapor temperature of at least 208° F. If the evaporator has forced circulation and an external calandria, 10 square feet of evaporating surface will be sufficient. The important thing here is that the holding capacity of the evaporator be sufficient to accommodate at least 200 gallons during operation. In the initial stages, evaporation can proceed concurrently with filtration. Evaporation should be started under vacuum, but toward the end, when the alcohol has been partially removed, it is desirable to operate under atmospheric pressure, since one of the objectives of this evaporation is to obtain the rutin in solution in the minimum amount of water. This cannot be feasibly done except at temperatures corresponding to atmospheric boiling. The purpose of attempting to have the smallest possible amount of water present at the end of the evaporation is to eliminate excessive loss of mother liquor, since this water will be the mother liquor from the first crystallization. Experience has shown that the approximately 26 pounds of rutin present at this stage would require about 600 gallons of pure water to put it into solution. However, if in the course of evaporation the rutin is not permitted to precipitate, it is easily possible to finish with the rutin in solution in as little as 200 gallons.

The concentrated hot aqueous solution is filtered through a preheated wooden filter press having about 30 square feet of filtering surface. A good clarifying paper supported by cloth should be used to dress the press. No filter aid is required. The 200-gallon batch can be filtered in about 30 minutes. The filtrate is discharged into one of two 300-gallon crystallizing tanks.

ALTERNATE PROCEDURE FOR CURD REMOVAL - The procedure just described for removing nonrutin solids in curd form is the one customarily employed when rutin free of alcohol insolubles is to be produced, the insolubles being subsequently removed with anhydrous alcohol. When a small amount of alcohol-insoluble material can be tolerated in the finished product, the alcohol-insoluble removal step can be eliminated by removing more of the insolubles in the curd-removal step. This is done by using a larger proportion of isopropyl alcohol. For example, if 2 volumes of 91 percent by volume isopropyl alcohol per volume of concentrate are used, the concentration of alcohol in the mixture is approximately 67 percent by volume. If 2-1/2 volumes of 99 percent isopropyl alcohol are used per volume of concentrate, the concentration of alcohol in the mixture will be about 77.5 percent by volume. Under the latter conditions, a much larger quantity of curd is removed and relatively pure rutin can be obtained by carrying out the succeeding steps in the usual way, but eliminating the alcohol-insoluble removal step.

CRYSTALLIZATION OF CRUDE RUTIN - The two 300-gallon crystallizing tanks may be made of wood. Each should have a stainless steel cooling coil and should be equipped with an agitator. As soon as the hot filtrate enters the crystallizing tank, cooling and agitation should be started. To facilitate crystallization, a few rutin crystals should be added for seed, after the solution has cooled sufficiently to prevent the seed from dissolving. The solution should be agitated at least periodically during the crystallization period, which should be at least 44 hours. Excessive periods of crystallization should not be used, as fermentation may develop, with consequent filtration difficulties.

On the completion of crystallization, the crude crystals are filtered off. It is good operating procedure to decant much of the liquor through the filter press before the crystals are stirred into suspension. This will shorten the time for filtration. The press can be of wood and should have a filtering surface of about 75 square feet. One-inch frames will be satisfactory. The filter medium can be fine twill cloth. At the completion of filtration, the crystals should be washed in the press with distilled water or tap water adjusted with sulfuric acid, if necessary to reduce the pH below 7. The residual liquor is then blown out of the press with air, and the rutin is removed and dried overnight at about 220° F. It should be possible to filter off one batch of crystals (about 30 pounds dry weight of crude rutin), wash them, and blow out the press in 3 hours.

REMOVAL OF ALCOHOL INSOLUBLES - Since the dried crude rutin can be satisfactorily stored, it would be economical to carry out the remaining operations only once a week. Hence, the batch from this point on will include all the crude rutin produced in 5 days of operation, that is, about 150 pounds. Since the standards of the Eastern Regional Research Laboratory have required that the finished product be free from material insoluble in anhydrous ethyl alcohol, these insolubles are removed before the final crystallization.

The 150 pounds of dried rutin is finely ground to insure rapid solution in alcohol. Either anhydrous ethyl alcohol or 99 percent by volume isopropyl alcohol may be used. Enough alcohol should be added to dissolve the pure rutin present when the alcohol is at its boiling point. Approximately 220 gallons of isopropyl or 150 gallons of ethyl alcohol will suffice. The tank used for this operation is the same one employed for precipitation of the curd. (Fig. 4, tank A). It should be thoroughly cleaned before being used for this step. The alcoholic solution is agitated, steam is introduced into the coils, and the solution is boiled for about 10 minutes. Water is then introduced into the coils, and the solution is cooled to about 80° F. and filtered.

A steam-jacketed stainless steel filter press with about 75 square feet of filtering surface is recommended. This is press D in the flow diagram (Fig. 4). The jacket is not used here, but will be required when the press is employed in a subsequent operation. The dressing should be clarifying paper backed with cloth. No filter aid is used. Filtration should be accomplished in about one-half hour. To remove the last traces of rutin from the insolubles, the cake is washed with alcohol of the same strength as that employed in dissolving the rutin and finally the press is blown out.

The filtrate from the press passes to the evaporator (Fig. 4, B) used to replace alcohol with water in the curd-removal step, but the evaporator should first be thoroughly boiled out with water to remove residual traces of curd. The evaporation is carried out under vacuum until practically all the alcohol is removed. Some water must be added during evaporation. The amount is unimportant, if there is enough to keep the rutin crystals in suspension, so that they can be pumped from the evaporator to the solution tank. The evaporator should be rinsed out with water in order that too much pure rutin may not be carried back into the process when the evaporator is used for its other function.

FINAL CRYSTALLIZATION AND DRYING - The solution tank should have a capacity of 3,000 gallons. It can be of wood and should be equipped with an agitator and stainless steel steam coil. Enough boiling filtered water to dissolve the rutin should be added to the rutin slurry from the evaporator. The water should be treated if necessary with sulfuric acid so that, on boiling, the pH will be below 7.

Associated with rutin in the buckwheat plant is red pigment, which in pure rutin is considered objectionable. Therefore, sufficient silica gel to adsorb this red pigment is added at this stage.[10] We have customarily used material passing a 20-mesh screen but held on a 65-mesh screen. The quantity of silica gel required depends not only on the individual lot of buckwheat but to some extent on the time involved in extracting the rutin from the meal. Long extraction times require larger quantities. The quantity required should first be established by a laboratory test. For the extraction cycle recommended here about 200 pounds will in general be required for a batch of this size, that is, the rutin from 4,650 pounds of meal. To adsorb the red pigment effectively, the silica gel must be intimately mixed and maintained in suspension in the boiling solution for at least 30 minutes. Since it is heavy, this will require strong agitation. The boiling period not only assures effective use of the silica gel, but it causes some nonrutin material to precipitate, thus giving a purer product on filtration.

The boiling solution is filtered through a steam-jacketed stainless steel filter press having about 75 square feet of filtering surface. This is the filter press used for filtering the alcohol insolubles (Fig. 4, D). A good clarifying paper backed with cloth should be used. No filter aid is required. Before the filtrate is discharged into the final crystallizing tank, it should be recycled to the dissolving tank until it is sparkling clear.

The final crystallizing tank should be of stainless steel and should be jacketed or equipped with a stainless steel cooling coil and an agitator. A capacity of 3,000 gallons is required. As soon as the clear filtrate begins to be discharged into this tank, cooling water should be turned on and the agitator started, to cool the filtrate rapidly. As soon as the solution is cool enough to prevent the rutin from dissolving, some seed crystals should be added, unless precipitation has already started. The crystallizing period should not be less than 16 hours or more than 20 hours. If the solution is allowed to stand too long, some nonrutins may precipitate with the pure rutin. The solution should be agitated periodically during the crystallizing period.

The pure rutin crystals are filtered off again with a stainless steel filter press designed to permit thorough washing of the cake. It should have 75 square feet of filtering surface, and should be dressed with clean, fine twill cloth. A separate filter press is specified for this filtration in order to prevent contamination of the pure rutin.

[10] J. F. Couch, C. F. Krewson, and W. L. Porter, Patent Application, Ser. No. 685,632.

At the completion of filtration, the rutin is washed in the press with distilled water, and the press is finally blown out with clean air. In the suggested cycle, adequate time is allowed to filter the rutin in several batches. The filtration rate will prove too slow if the entire batch is filtered in one operation. The filtrate and the wash waters from this operation are accumulated in a 3,000-gallon wooden storage tank having means for introducing open steam. These liquors contain significant amounts of rutin and are used in extracting the meal.

The pure rutin should be brought to dryness at 220° F. The unit used for drying the crude rutin can be used for this. If a forced-circulation drier is used, care must be taken to avoid contamination of the product by dirt in the air.

YIELD OF RUTIN - If the operations have been properly carried out, approximately 100 pounds of pure rutin should be obtained from the week's operation. This would correspond to a yield of 78 percent of the rutin in the meal. Careful pilot-plant operations have given yields of slightly more than 80 percent. Factors contributing to good yield are thorough washing of the curd in the filter press, complete crystallization, which is obtained by good techniques, and avoidance of prolonged heating of rutin in alcohol or water.

Typical Operating Cycle

In order to illustrate how proper scheduling will make it possible for the same equipment to be used for several operations, an operating cycle is shown in Table 1.

Three 310-pound batches of meal are extracted each 24-hour day. During the next 8-hour day, the curd is removed, and the crude crystallization started. Forty-four hours later, the crude rutin is filtered off during the day shift and dried overnight. Once a week--during the day shift on Monday--the accumulated dry crude rutin is treated to remove alcohol-insolubles, and laid down to crystallize. The pure rutin is then filtered off during the day shift on Tuesday. Thus, some stage of rutin production is being carried out on each of 300 days in the year. Certain operations, however, are carried out only periodically. For example, extraction is done 24-hours per day for 250 days; curd is removed 8 hours per day for 250 days; and alcohol-insolubles are removed only once a week in an 8-hour operation. For maximum utilization of equipment and for convenience in maintaining crystallization cycles, the operations should be spread over 300 days.

Suggested List of Equipment

Extraction tank: Wooden tank, 9 feet in diameter by 7 feet deep, with a false bottom covered with 30-mesh stainless steel screen. Equipped with a slow-speed sweep agitator and with a steam sparger under the screen, both of stainless steel.

Extract draw-off pump: Stainless steel positive-delivery rotary pump. Must discharge 20 gallons of hot extract per minute.

Storage tank for extracts: Wooden tank, 5 feet in diameter by 5 feet deep, equipped with agitator and stainless steel steam coil. Capacity, 700 gallons.

Filter press for extracts: Wooden filter press with 60 square feet of filtering surface.

Vacuum evaporator for concentration of extracts: Stainless steel vacuum evaporator with jet condenser. Must evaporate approximately 150 gallons of water per hour. External calandria with 25 square feet of evaporating surface.

Coagulation tank: Stainless steel tank, 3-1/2 feet in diameter by 4-1/2 feet deep, equipped with agitator, reflux condenser, and jacket for steam or cooling water. Capacity, 300 gallons. Used also for removal of alcohol insolubles.

Filter press for curd: Wooden filter press with 2-inch frames and 75 square feet of filtering surface.

Evaporator for removal of alcohol: Stainless steel vacuum evaporator (condenser and receiver may be copper). External calandria of 10 square feet of evaporating surface. Holding capacity, 200 gallons. Used after both removal of curd and removal of alcohol insolubles.

Filter press for hot filtration: Wooden filter press with 30 square feet of filtering surface.

First crystallizing tanks: Two wooden tanks, 3-1/2 feet in diameter by 4-1/2 feet deep, equipped with agitators and stainless steel cooling coils. Capacity, 300 gallons each.

Crystal filter press: Wooden filter press with 75 square feet of filtering surface. To filter off crude rutin.

Tray drier: Tray drier to dry both crude and pure rutin. Must be capable of evaporating 200 pounds of water from 300 pounds of wet cake in 40 hours.

Filter press for removing alcohol insolubles: Stainless steel steam-jacketed filter press with 75 square feet of filtering surface. Used also for filtering hot-water solution of rutin before final crystallization.

Solution tank: Wooden tank, 7-1/2 feet in diameter by 9 feet deep, equipped with stainless steel steam coil and agitator sufficiently strong to keep silica gel thoroughly mixed. Capacity, 3000 gallons.

Final crystallizing tank: Stainless steel tank, 7-1/2 feet in diameter by 9 feet deep, equipped with agitator and jacket for cooling water. Capacity, 3000 gallons.

Final crystal filter press: Stainless steel backwash filter press with 75 square feet of filtering surface.

Storage tank for mother liquor: Wooden tank, 7-1/2 feet in diameter by 9 feet deep, with open steam jet for heating. Capacity, 3,000 gallons.

Hot-water supply system: System capable of supplying 500 gallons of boiling water at 2-1/2 hour intervals.

Rectification still: A still capable of rectifying about 250 gallons of approximately 75 percent alcohol to 91 percent alcohol per day.

Storage tank for rectified alcohol: Wooden tank, 4-1/2 feet in diameter by 4-1/2 feet deep. Capacity, 500 gallons.

Applicability of Process to Green Plant

The question naturally arises as to the possibility of extracting green buckwheat with hot water. Exploratory tests indicate that the yields may be lower than those obtained with the dried plant, and the rutin is more difficult to purify. However, the work has not been carried far enough at the Laboratory to establish the feasibility of such a process. It is reported that the extraction of green buckwheat with hot water has been carried out with commercial success.

DILUTE-ALCOHOL EXTRACTION PROCESS

General Description

Buckwheat meal, either leaf meal or whole meal, may also be extracted with dilute alcohol to remove the rutin. In this extraction, the meal is given three 12-hour extractions at room temperature, followed by a short wash with alcohol of the same strength. The extracts are then filtered and concentrated by evaporation. Tarry materials, such as fats and resins, which are extracted from the meal by dilute alcohol, are removed by boiling to coalesce them, followed by filtration.[11] Crude rutin is obtained by allowing the filtrate to crystallize overnight. It is then filtered off, dried, and given a short recrystallization.

Figure 5 is a flow diagram for the production of rutin by this process.

Details of Operation

The operations described here are those required in a plant producing 100 pounds per week of pure dry rutin from buckwheat meal containing 3 percent rutin on the moisture-free basis and having a moisture content of 8 percent. A rutin yield of 78 percent, based on the rutin content of the meal, is assumed. This scale of operations will produce 5,000 pounds of pure rutin in an operating year of 300 days. The equipment described is that required in a new factory designed for the express purpose of producing rutin. It is recognized however that in most cases rutin will be produced in equipment already available in pharmaceutical plants, since much of such equipment will undoubtedly be adaptable to the rutin process. In many cases wooden tanks and wooden filter presses are specified. This represents the cheapest type of equipment that could be employed. When stainless steel equipment is available, it could be preferably employed. When stainless steel equipment has been specified, it must be used, as less corrosion-resistant materials may contribute impurities to the finished rutin. Even traces of dissolved iron or copper are objectionable.

[11] Roderick K. Eskew, patent application, Serial No. 689,348

Table 1. TYPICAL OPERATING CYCLE FOR HOT-WATER EXTRACTION PROCESS

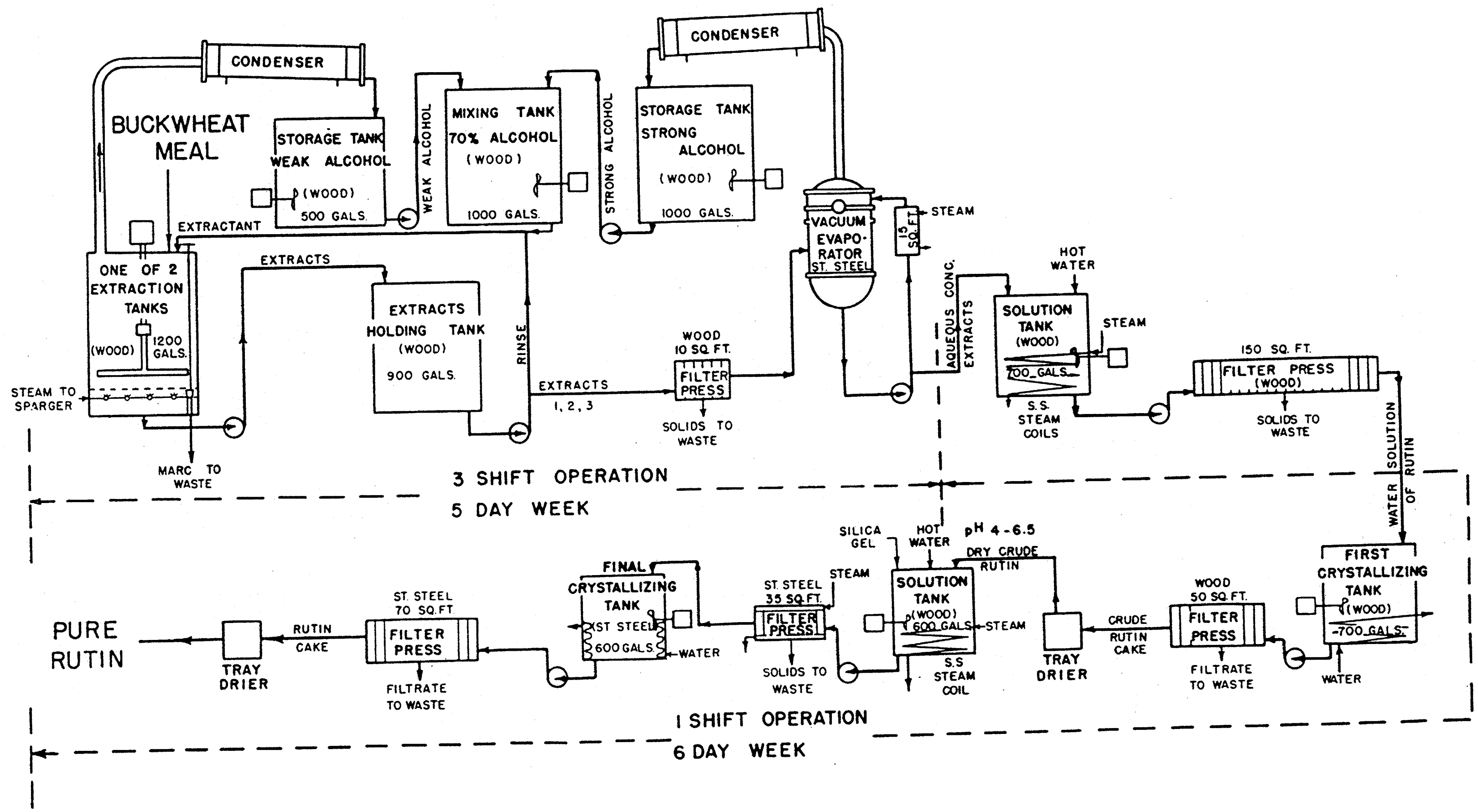
Day	Time	Extract meal and concentrate extracts Batch no.	Remove curd and start crystal-lization Batch no.	Filter off crude rutin Batch no.	Dry crude rutin Batch no.	Remove alcohol-insolubles and start crystal-lization Batch no.	Filter off pure rutin and start drying Batch no.
Thursday	8 a.m. - 4 p.m.	1					
	4 p.m. - 12 m.	2					
Friday	12 m. - 8 a.m.	3					
	8 a.m. - 4 p.m.	4	1-3				
	4 p.m. - 12 m.	5					
Saturday	12 m. - 8 a.m.	6					
	8 a.m. - 4 p.m.		4-6				
Sunday							
Monday	8 a.m. - 4 p.m.	7		1-6			
	4 p.m. - 12 m.	8			1-6		
Tuesday	12 m. - 8 a.m.	9			1-6		
	8 a.m. - 4 p.m.	10	7-9		1-6		
	4 p.m. - 12 m.	11					
Wednesday	12 m. - 8 a.m.	12					
	8 a.m. 4 p.m.	13	10-12				
	4 p.m. - 12 m.	14					
Thursday	12 m. - 8 a.m.	15					
	8 a.m. - 4 p.m.	16	13-15	7-9			
	4 p.m. - 12 m.	17			7-9		
Friday	12 m. - 8 a.m.	18			7-9		
	8 a.m. - 4 p.m.	19	16-18	10-12			
	4 p.m. - 12 m.	20			10-12		
Saturday	12 m. - 8 a.m.	21			10-12		
	8 a.m. - 4 p.m.		19-21	13-15			
Sunday					13-15		
Monday	8 a.m. - 4 p.m.	22		16-21		1-15[1]	
	4 p.m. - 12 m.	23			16-21		
Tuesday	12 m. - 8 a.m.	24			16-21		
	8 a.m. - 4 p.m.	25	22-24		16-21		1-15
	4 p.m. - 12 m.	26					

1 Crude rutin accumulated for 1 week

FIG. 5

FLOW DIAGRAM FOR PRODUCTION OF RUTIN FROM BUCKWHEAT MEAL

BY DILUTE ALCOHOL PROCESS



EXTRACTION - Two wooden tanks, each having a capacity of 1,200 gallons are required for the extraction. Each tank should be equipped with a false bottom covered with 30-mesh stainless steel screen, below which is a steam sparger for the introduction of open steam to be used in stripping the alcohol from the marc at the end of each extraction cycle. The tanks should be tightly covered and supplied with vapor connections to a condenser. The recovered alcohol is stored for reuse in a wooden tank of 500 gallons capacity. Both tanks should be connected to a pump for discharge of the extracts to a 900-gallon wooden holding tank.

Either mechanical agitation or recirculation of the liquor during extraction is necessary if the extraction cycle is to be kept short. Studies on extraction rates have shown that with adequate circulation, equilibrium can be reached in 8 hours. Because of variations in the physical condition of buckwheat meal, however, a 12-hour extraction cycle is recommended. With a ratio of 1 gallon of alcohol to 1 pound of air-dry meal, three 12-hour extractions and one wash removes about 97 percent of the rutin.

The operation of the two extraction tanks is staggered. Each should be charged with 930 pounds of meal and 930 gallons of alcohol. Pilot-plant experiments have shown that approximately 70 percent by volume of ethyl alcohol is a good concentration to use and is a much better extracting agent than 95 percent.[12] Benzol-denatured alcohol is also satisfactory. The first three extracts from each batch are concentrated and carried on through the process; the fourth extract or wash, which is allowed to stay on the meal only half an hour, is reused as the first extractant in a succeeding batch. For effective removal of rutin, at least 66 percent of the extractant should be drawn off in each extraction, that is, about 614 gallons. The 1-1/2-hour period called for in the suggested operating cycle should be adequate for this draw-off. At the completion of an extraction cycle, much of the alcohol can be recovered from the marc by introducing open steam under the false bottom and condensing the vapors driven off.

CONCENTRATION OF EXTRACTS - As rapidly as the extracts accumulate in the holding tank, they are filtered and evaporated. A wooden filter press with an area of 10 square feet should be adequate, as filtration is extremely rapid. No filter aid is needed. A fast filter paper backed with twill cloth is used. One-inch frames in the press should have adequate holding capacity for the solids filtered out unless the meal is exceptionally fine, in which case a larger holding capacity should be provided, either by the use of 2-inch frames or a larger number of frames. The three extracts are filtered, but the rinse may be reused without filtration.

12 Edward L. Griffin, Jr. U.S. Patent 2,425,094. Assigned to the Secretary of Agriculture.

Through proper staggering of the extraction cycles, a first, second, and third extract, although not from the same extraction tank, will be available during each 24-hour period and may be combined and evaporated to form one batch. Such a batch would contain all the rutin extracted from 930 pounds of meal. The extracts are concentrated in a stainless steel vacuum evaporator. If a forced circulation, external calandria-type evaporator is used, 15 square feet of heating surface will be sufficient, but if a vacuum pan is used, an increase in the heating surface will be required to achieve the same evaporation rate. The evaporator must be capable of reducing approximately 1840 gallons of extract to about 150 gallons of concentrate in a total of 18 hours. This concentration should be carried out in two or three portions in order to minimize the time of holding the rutin in the evaporator. The purpose of this concentration is to remove the alcohol and reduce the volume to less than that required to dissolve the rutin present. When the alcohol has been driven off, the residual concentrate contains a slurry of rutin crystals.

REMOVAL OF TARRY SUBSTANCES - The concentrate is transferred to a 700-gallon wooden solution tank with a stainless steel heating coil. Sufficient hot water is added to dissolve the rutin. This will require a total of approximately 23 gallons of hot water per pound of rutin present. The solution is boiled for 30 minutes. Tarry substances are coalesced by the concentration in the evaporator and the subsequent boiling, and they can be eliminated by thorough filtration. This is done in a wooden filter press having about 150 square feet of filtering surface. It should be dressed with a dense filter pad, such as the Republic Filter Corporation's "Serum" paper[13] backed with twill cloth. The press should be preheated with hot water. The solution is recycled until the filtrate is sparkling clear. The filtrate is then discharged into the crystallizing tanks. Couch and Krewson observed that filtration could be facilitated by first straining the solution through a mat of glass wool.[14]

This purification process depends on thorough removal of tarry material at this stage, hence extremely fine filtration is essential. Care should be taken not to contaminate the rutin at a later stage by reuse of equipment fouled with tars.

CRYSTALLIZATION OF CRUDE RUTIN - The crystallizing tank, which may be of wood, should be equipped with stainless steel cooling coils and have 700 gallon capacity. As soon as the hot filtrate enters the crystallization tank, cooling and agitation should be started to facilitate crystallization. After the solution has cooled sufficiently to prevent the rutin from dissolving, a few rutin crystals should be added for seed. The tank should be agitated at least periodically during the crystallization period, which should be about 15 hours. The crystallizing period should not be excessive, as fermentation may develop with

13 The mention of commercial products does not imply that they are endorsed or recommended by the Department of Agriculture over others of a similar nature not mentioned.

14 J. F. Couch and C. F. Krewson. Patent Application, Case No. 2240.

consequent filtration difficulties. Furthermore, when crystallization is continued unduly, nonrutins precipitate, necessitating a more stringent final purification for their removal.

When crystallization is complete, the crude crystals are filtered off. It is good operating procedure to decant much of the liquor through the filter press before the crystals are stirred into suspension. This will shorten the time for filtration. The press can be of wood and should have a filtering surface of 50 square feet. One-inch frames will be satisfactory. The filter medium should be fine twill cloth. At the completion of the filtration, the crystals should be washed in the press with distilled water or tap water adjusted with sulfuric acid, if necessary to reduce the pH below 7. The residual liquor is then blown out of the press with air, the rutin is removed and dried overnight at about 220° F. It should be possible to filter off one batch of crystals (about 30 pounds, dry weight, of crude rutin), wash them, and blow out the press in 3 hours.

FINAL CRYSTALLIZATION AND DRYING - The dried rutin is dissolved in boiling filtered water, treated with sulfuric acid if necessary to reduce the pH below 7.0. Approximately 16 gallons of boiling water will be required per pound of crude rutin. This operation can be carried out in a 600-gallon wooden tank, equipped with an agitator and a stainless steel steam coil.

Associated with rutin in the buckwheat plant is red pigment, which in pure rutin is considered objectionable. Therefore sufficient silica gel to adsorb this red pigment is added at this stage. Since the quantity of silica gel required depends on the individual lot of buckwheat, it should first be determined by a laboratory test. About 10 pounds may be required for a batch of this size (that is, the rutin from 930 pounds of meal). To adsorb the red pigment effectively, the silica gel must be intimately mixed and maintained in suspension in the boiling solution for at least 30 minutes. Since it is heavy material, this will require strong agitation. The boiling period not only assures effective use of the silica gel, but it causes the last traces of tarry materials to coalesce, so they may be completely removed in the succeeding filtration.

The boiling solution is filtered in a steam-jacketed stainless steel press having about 35 square feet of filtering surface. Here again "Serum" pads backed with twill cloth are used. Before the filtrate is discharged into the final crystallizing tank, it should be recycled to the dissolving tank until it is sparkling clear.

The final crystallizing tank should be of stainless steel and should be jacketed or equipped with a stainless steel cooling coil and an agitator. A capacity of 600 gallons is required. As soon as the clear filtrate begins to be discharged into this tank, cooling water should be turned on and the agitator started, so that the filtrate may be cooled rapidly. As soon as the solution is cool enough to prevent the rutin from dissolving, some seed crystals should be added, unless precipitation has already started. Slow, continuous agitation should be provided during crystallization.

The time allowed for final crystallization is important. Pilot-plant experience has shown the rutin to be so pure at this stage that crystallization is essentially complete 4 hours after the temperature of the solution has reached 100° F. We recommend that after 4 hours, the rutin be filtered off on a 70-square foot stainless steel filter press having backwash channels and dressed with fine twill cloth. It is thoroughly washed in the filter press with distilled water, blown out with clean air, and dried at 220° F. This rutin should be free from alcohol-insoluble material and of high purity.

In the foregoing process, about 78 percent of the rutin in the meal should be recovered.

The alcohol-extraction process depends on a high degree of coagulation and removal of tarry material and nonrutins and also on carefully controlled crystallization conditions. Deviation from the procedure may result in the presence of alcohol-insoluble material in the final rutin. If these are present, however, they may be removed by the same procedure described under "Removal of Insoluble Material" and "Final Crystallization and Drying" in the hot-water extraction process.

Typical Operating Cycle

In order to illustrate this process, an operating cycle is shown in Table 2.

In each of two separate tanks, a 930-pound batch of meal is extracted for 48 hours. The extraction cycles are staggered so that a first, second, and third extract are available during each 24-hour period but not all from the same tank. These three extracts are combined and concentrated together. During the next 8-hour day, they are redissolved, and filtered, and the crude crystallization is started. The crude rutin is filtered off on the following day and dried. On the fourth day, it is recrystallized, and when dry yields pure rutin.

Extraction and concentration are carried out on a 24-hour basis 5 days a week, but all other operations except drying can be conducted on an 8-hour basis 5 days a week. For convenience in maintaining crystallization cycles, some operation of rutin production is in progress 300 days a year, but each individual operation is carried out only 250 days a year, 5 days a week.

Suggested List of Equipment

Extraction tanks: Two wooden tanks, each 6 feet in diameter by 6 feet deep, with false bottom covered with 30-mesh stainless steel screen. Capacity, 1,200 gallons each. Equipped with steam sparger under false bottom, and tightly fitting cover. One condenser serves both tanks when alcohol is stripped from the marc.

Extract draw-off pump: Stainless steel positive-delivery rotary pump. Must discharge 15 gallons of dilute alcohol per minute.

Holding tank for extracts: Wooden tank, 5 feet in diameter by 6 feet deep. Capacity, 900 gallons.

Table 2

TYPICAL OPERATING CYCLE FOR ALCOHOL-EXTRACTION PROCESS

	24-hour operation ending 8 a.m.		Operations (except drying) during day shift only, 8 a.m. - 4 p.m.				
	Extract meal and concentrate extracts						
Day	Tank 1 Extract No.	Tank 2 Extract No.	Remove tars and start crystal-lization Batch No.	Filter off crude rutin Batch No.	Dry crude rutin Batch No.	Recrystal-lize crude rutin and filter off pure rutin Batch No.	Dry pure rutin Batch No.
Wednesday	1	2 and 3					
Thursday	2 and 3	1	1[1]				
Friday	1	2 and 3	2[1]	1	1		
Saturday			3[1]	2	2		
Sunday							
Monday	2 and 3	1		3	3	1	1
Tuesday	1	2 and 3	4[1]			2	2
Wednesday	2 and 3	1	5[1]	4	4	3	3
Thursday	1	2 and 3	6[1]	5	5	4	4
Friday	2 and 3	1	7[1]	6	6	5	5
Saturday			8[1]	7	7		
Sunday							

1 Total concentrate (three extracts) from previous day's operation

Evaporator: Stainless steel evaporator with 15 square feet of evaporating surface in an external calandria. Must be able to evaporate 1,635 gallons of 70 percent alcohol in 18 hours and must have a holding capacity of 100 gallons.

Filter press for extracts: Wooden filter press with 10 square feet of filtering surface.

First solution tank: Wooden tank, 5 feet in diameter by 5 feet deep, equipped with agitator and stainless steel steam coil. Used to dissolve rutin and coagulate tars. Capacity, 700 gallons.

Filter press for removing tars: Wooden filter press with 150 square feet of filtering surface.

First crystallizing tank: Wooden tank, 5 feet in diameter by 5 feet deep, equipped with agitator and stainless steel cooling coils. Capacity, 700 gallons.

Crystal filter press: Wooden filter press with 50 square feet of filtering surface. To filter off crude rutin crystals.

Tray drier: A tray drier capable of evaporating about 60 pounds of water from 90 pounds of wet cake per day.

Second solution tank: Wooden tank, 4-1/2 feet in diameter by 5 feet deep, equipped with agitator and stainless steel steam coil. Capacity, 600 gallons. Used for dissolving crude rutin and treating with silica gel.

Filter press for second hot filtration: Stainless steel jacketed filter press having a filtering area of 35 square feet.

Tank for final crystallization: Stainless steel tank, 4-1/2 feet in diameter by 5 feet deep, equipped with agitator and having a jacket for cooling. Capacity, 600 gallons.

Crystal filter press: Stainless steel backwash filter press having a filtering surface of 70 square feet.

Tray drier: A tray drier capable of evaporating 40 pounds of water from 60 pounds of wet cake per day.

Tank for preparing 70 percent alcohol: Wooden tank, 5 feet in diameter by 7 feet deep, equipped with agitator. Capacity, 1,000 gallons.

Storage tank for alcohol recovered from evaporation of leaches: Wooden tank, 5 feet in diameter by 7 feet deep, equipped with agitator. Capacity, 1,000 gallons.

Storage tank for weak alcohol recovered from marc: Wooden tank, 4-1/2 feet in diameter by 5 feet deep, equipped with agitator. Capacity, 500 gallons.